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Review comments on "Draft sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4"

Submitted by Greg Meeker, USGS, 29 April, 2002

General Comments My comments on this document and procedures referenced within are primarily directed at analytical techniques and mineralogy Comments regarding the validity of sampling methods will be submitted by others from USGS I do not comment here on sample control issues such as labeling shipping storage, safety, etc which comprise a significant portion of this document

In general, the document is impressive in its bulk but seems light on technical content Much of this detail is apparently included in other SOPs, one of which I have (SEM SOP) and have reviewed below Others such as the IR SOP and the Laboratory Training Outline I do not have and am therefore unable to comment on at this time

My overall impression is that the document was hastily prepared without adequate knowledge of mineralogy, geology, asbestos terminology and analytical procedures Many of the sections, such as the IR and SEM analytical sections, are inappropriately duplicated, reflect a lack of knowledge of the techniques, and are inappropriate for this specific problem References to standards and standardization procedures appear to be "boiler plate", and reflect a lack of technical knowledge

Of particular concern are the following 1) Misuse of terminology, which will present an easy target for anyone looking to find fault with the methods outlined in the document, 2) Inappropriate use of standards and use of incorrect standards, 3) inappropriate analytical conditions and 4) incorrect equations

Finally, for the most part I have not commented on sample preparation techniques and offer the USGS sample preparation procedure that is in preparation as an alternative

Listed below are specific comments, in some cases I have suggested changes

1] Page (pp) 1-1, paragraph (P) 5, last line *The major concern of the LAA is the content of tremolite* This sentence uses improper terminology and is too restrictive If the major concern is asbestos then that should suffice Delete this sentence

2] Page 1-2, P 5 *tremolite rocks* change to **amphibole-rich rocks**

3] pp 2-1, P 2, *This ore body contained amphibole asbestos fibers* change to **This ore body contained amphibole asbestos fibers with compositions including tremolite, actinolite, winchite, and richterite as defined by (Leake, 1997)**

4] pp 2-1, P 3, *This process boiled the water trapped* change to **This process explosively vaporizes the water contained within the mica structure causing the vermiculite to expand by a factor of 10 to 15**

5] pp 2-2, P 5, Change *tremolite rocks* to **LAA-containing rocks**

6] pp 2-3, P 4, change *tremolite asbestos the form associated with the mined* to **tremolite and actinolite asbestos, varieties commonly associated with**

7] pp 3-1, P 2, change *tremolite rocks* to **LAA-containing rocks**

8] pp 3-6, second line, change *Visually inspect land use areas for visible vermiculite* to **Visually inspect land use areas for vermiculite or LAA-containing rock**

9] pp 3-7, P 7, Comment Where is the I R method?

10] pp 4-4, P 1, Comment 0-1 inch sample depth is too shallow

11] pp 6-5, P 7, Comment EPA does not make or provide calibration standards for SEM, EDS or IR, delete this sentence

12] pp 9-1, Comment Add reference Leake, et al , 1997, Report

13] Appendix A (A), IR method, pp 2, Equation Change *Found* to **Measured** change *True* to **actual**

14] A, IR method, pp 3, P 6, 1) The term asbestiform refers to the physical form of a particle, i e crystallized with the form of asbestos It does not refer to different asbestos phases such as tremolite, actinolite, etc , nor does the term only apply to asbestos minerals This usage shows a serious misunderstanding of asbestos terminology and compromises the integrity of the entire document 2) Laboratory Control Samples LCSs are used to check the calibration, not the accuracy of the calibration standards 3) IR cannot count or size fibers 4) Equation at bottom of page change *Found* to **Measured** and *True* to **Actual**

15] A, IR method, pp 4, Section 4 1, I don't see the point of Laboratory Duplicate Samples as defined here The sample preparation as described in this method will not produce a homogeneous sample, therefore, this control will not work

16] A, IR method, pp 5, Section 4 2, Do to the heterogeneous nature of LAA found in the field, it will not be possible, with confidience, to obtain two field samples with consistant LAA concentrations These should instead be prepared standards submitted as blinds

17] A, IR method, pp 5, Section 4 3, Same as above

18] A, SEM method, pp 2, P 1, Comment This is not the IR method

19] A, SEM method, pp 3, P 1, 1) 175 eV is a very poor resolution for modern EDS detectors, a value of 145 eV (for Mn Kα) would be more appropriate 2) The peak resolution must be specified at specific energies, this is usually done with Mn Kα and F Kα

20] A, SEM method, pp 3, Section 1 1 4, EDS manufactures normally do not have a criterion for Na sensivity

21] A, SEM method, pp 3, section 1 2, Change equation as specified above

22] A, SEM method, pp 4-7, See comments from IR section

23] Appendix D, General comment I am concerned about this section and the details that are lacking regarding identification criteria (see comment 34, #6), and the information that is being given out by the "Lab Mentors" The fibrous amphibole found in Libby is unusual when compared to "normal" commercial asbestos, but it is *not unique* Care must be taken not to imply that Libby-type amphiboles (asbestos) are a distinct class of minerals, and that the names "Libby-type amphibole" or "Libby amphibole asbestos" (LAA) are used only as an analytical aid and do not creep into regulatory language Regulatory terminology is confusing enough as it is I believe it would be a mistake to add to the confusion by introducing more terminology that does not have a precise mineralogical definition

24] Magnesio-arfvedsonite does not appear to occur in fibrous form in the Libby material

Review comments on "Asbestos analysis of soil by scanning electron microscopy and energy dispersive x-ray spectroscopy EPA-LIBBY-01"

25] Section 2 1, Why is this method not applicable to samples containing talc?

26] Section 5 0, SRMs 1866 and 1867 are not appropriate for testing peak resolution This should be done with **polished** peak resolution standards such as Mn and CaF at the appropriate energies See 19 above

27] Section 6 2, The "squeeeze test" is not appropriate for this SOP Different materials will behave differently regardless of water content A better method would be to test for weight loss over a specified time period

Section 7 1, See comment 14 above

28] Section 7 3 Mn standard need not be from NIST (NBS), any pure Mn standard is fine

29] Section 7 4, Magnification Calibration, +/- 5% is probably too tight for this method, +/- 10% should suffice In addition to magnification the aspect ratio should be checked

30] Section 7 4, Peak Centroid, Many of the newer instruments don't use Al-Cu This should be done according to manufacturers specifications

31] Section 7 4, Resolution, See comment 19 above

32] Section 7 4, Sodium Sensitivity, Crocidolite is the wrong standard for this test This test should be performed on a polished standard of known composition such as an albite or jadite Light element sensitivity from particles such as crocidolite will be affected by particle size, shape, composition, and substrate contribution

33] Section 7 5, Laboratory control samples, A laboratory control sample for evaluating calibration of the EDS detctor (and for inter-laboratory comparisons) should not be composed of fibers or particles It should be a polished, well characterized, calibration standard similar in composition to the unknowns being analyzed, i e amphibole A good LCS would be USGS BIR-1G There are many other appropriate materials available for LCSs

34] Section 7 6, Analysis procedures, 1) Operating voltage of 25 kV is too high to obtain good EDS results on small asbestos fibers, 2) Au should not be used for coating the samples, 3) Measurement at 10X in the SEM is a waste of time This should be done with a stereo microscope, 4) What does "asbestos nature" mean? 5) Lower limit for EDS detection depends on the operating voltage and beam current, 6) The statement that richterite can be differentiated from actinolite and tremolite by the presence of Na and K is wrong Both tremolite and actinolite can contain Na and K The only generally accepted way to determine the difference between the phases using electron microbeam techniques is by *quantitative* chemical analysis and by determining the crystal site chemistry as described in Leake, 1997, 7) The sample loading method described will cause charging and create conditions that will provide inconsistent results

35] Section 9 0, These equations are not correct The result is not cumulative, it is a percentage If each individual size fraction has 20% then the total for the entire sample should be 20%, the equation gives a total of 59% If the sample is 100% asbestos the equation gives a result of -200% In general, I don't think this type of approach is necessary for this stage of evaluation as we understand it from Jim Christiansen, a visual estimate based on the PE standards should be sufficient if the samples are properly prepared

36] Section 9 1, A calculation for "flakes" is unnecessary This was based on an incorrect assumption that flakes will contain abundant amphibole within the structure Flakes should be treated the same as other particles

37] Section 10 1, Field Duplicates, See 17 above

38] Section 10 1, Laboratory Duplicates, See 15 above

39] Sections 10 2 and 10 3, See 14

40] The EDS spectra should include type of detector i e Be window, thin window, etc , the primary accelerating voltage, and also a reference spectrum of a polished standard such as USGS BIR-1G